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Fourth Quarterly Progress Report
ULTRA-FINE PARTICLE SIZE FERROELECTRICS
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Abstract

This report describes work done during the fourth quarterly contract period for the project "Ultra-Fine Particle Size Ferroelectrics" which is directed to the preparation of controlled particle size polycrystalline dielectric films of ferroelectric materials; and also to theoretical studies of the phenomena associated with the changes in electrical properties which occur when the particle size of ferroelectric materials is reduced below certain levels.

During this program period, formulations comprising fine homodisperse solid solutions of calcium and barium titanates were prepared by a modification of the method of pyrolysis of the oxalate. When materials containing up to 10 mole percent substitution of barium by calcium were densified into ceramics, the qualitative effects on the dielectric constant were only minor when compared with pure barium titanate of a similar grain size; however, substantial improvements in dissipation factor were obtained, particularly at temperatures above 100°C.

Studies were directed to the preparation of dense films of barium titanate of the order of 10 microns thick, by the pyrolysis, in situ, of mixed organic compounds of barium and titanium on various electroded substrates. With the aid of direct x-ray diffraction analysis of the films, the formation of barium titanate was followed as a function of the firing temperature. It was found that coherent polycrystalline films of pure $BaTiO_3$ could be formed at temperatures as low as 1100°F, having crystallite sizes less than 0.5 microns, and containing no additives to inhibit grain growth.

Equipment for measuring polarization as a function of temperature, using the pyroelectric technique, was assembled and standarized against conventionally fired and polarized barium titanate specimens.

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1. MIXED CALCIUM AND BARIUM TITANATES

1.1 Preparation of Materials

Since x-ray fluorescence analysis indicated the presence of indeterminate amounts of calcium in the highest purity grade of barium titanate obtained, the starting materials used to produce high purity solid solutions were reagent grades of barium and calcium carbonates (Mallinkrodt), and reagent titanium dioxide (Fisher). The calculated stoichiometric amounts of these materials were dry blended, pressed into two inch diameter slugs, and calcined several hours at 2200°F. They were then disintegrated, repressed and recalcined.

The resulting coarse-particled material was converted to the mixed oxalate by digesting it in a 50% water solution of a 10% equivalent excess of oxalic acid; the digestion was performed under gentle reflux for 8 hours. After cooling, the solid was removed by filtration, washed with cold water several times and dried at 110°C.

Since preliminary studies indicated that if the digestion were carried out at too low a temperature or for too short a period of time, a depletion of titanium as high as 5% or more occurred, it was checked that the reaction conditions described above a product identical in stoichiometry to the starting material. First of all, thefiltrate and wash waters were combined, evaporated to dryness, calcined at 1500°F, and the residue calculated as TiO_2. The result obtained was that the titanium depletion thus estimated was less than 0.5%.

Secondly, a comparison of the Ti/Ba x-ray fluorescence intensity ratios for the starting solid solutions and the material obtained by conversion

to the oxalate and subsequent pyrolysis, indicated that no significant change in mole ratio had occurred. (Due to the low atomic number of calcium and consequent low intensity of its fluorescence radiation, it was not possible to obtain ratios sufficiently accurate for this analysis.)

Finally, x-ray diffraction indicated the presence of a single phase in the final reaction product.

The product of the pyrolysis of the oxalate of $Ca_{0.10}$ $Ba_{0.90}$ TiO_3 at 1500°F for 2 hours was analysed for particle size by direct electron microscopy, and was found to range from 100Å - 200Å , with an average at about 120Å

1.2 Electrical Properties of Fine-Grained Ceramic Ca-Ba-TiO_3

In Figure 1, the dielectric constant K is plotted against temperature for bodies of the base composition. $Ca_{0.10}$ $Ba_{0.90}$ TiO_3, prepared from starting powders with an average particle 120A°. The bodies contained 0.5% SiO_2 applied as Ludox AS; body number 38 contains 2.5% , and number 41, 0% Ta_2O_5 applied as a tantalum oxalate solution, according to the method detailed in the previous quarterly report of this project. The bodies were pressed at 10,000 psi, with no binder, baked out at 1500°F for 20 minutes, and fired for 20 minutes at 2700°F. Porosity was measured and found to be negligible.

The resulting grain sizes were found to average 12 and 0.25 microns when the amounts of Ta_2O_5 , employed as a grain growth inhibitor, were 0 and 2.5% by weight, respectively. Due to the very high purity of the starting materials, it was found necessary to add the SiO_2 to facilitate sintering,

despite the fact that the $CaTiO_3$ - $BaTiO_3$ eutectic melts about 70°F lower than pure $BaTiO_3$ (1).

In Figure 2, for purposes of comparison, the dielectric constant of the composition $BaTiO_3$ (derived from the oxalate) + 0.5% SiO_2 + 2.5% Ta_2O_5, is plotted. The body represented has an average grain size of 0.2 microns.

The graph of Figure 1, body 38, shows the reduced variation of dielectric constant with temperature typical of the bodies studied to date with particle sizes below about 0.5 microns. However, at this writing it is not known whether either of the two apparent maxima represents a Curie point; this information should become apparent from a study of the pyroelectric effect as a function of temperature.

Of particular interest is the dramatic reduction of the dissipation factor found in the bodies containing the 10 mole percent calcium substitution. As a typical example, in Figure 3 the dissipation factor is plotted against temperature for the bodies $Ca_{0.10}Ba_{0.90}TiO_3$ + 0.5% SiO_2 + 2.5% Ta_2O_5, and $BaTiO_3$ + 0.5% SiO_2 + 2.5% Ta_2O_5. The reduced power losses resulting from the calcium sibstitution may be ascribed principally to an increase in resistivity, since the dissipation factor remains low above about 100°C.

In summary, the effect of calcium substitution in a fine-particled barium titanate ceramic appears to be that the general behavior of the dielectric constant does not change radically, but that there is a significant reduction in dissipation factor.

FIGURE 1: Dielectric Constants of $Ca_{0.10}Ba_{0.90}TiO_3$ Bodies

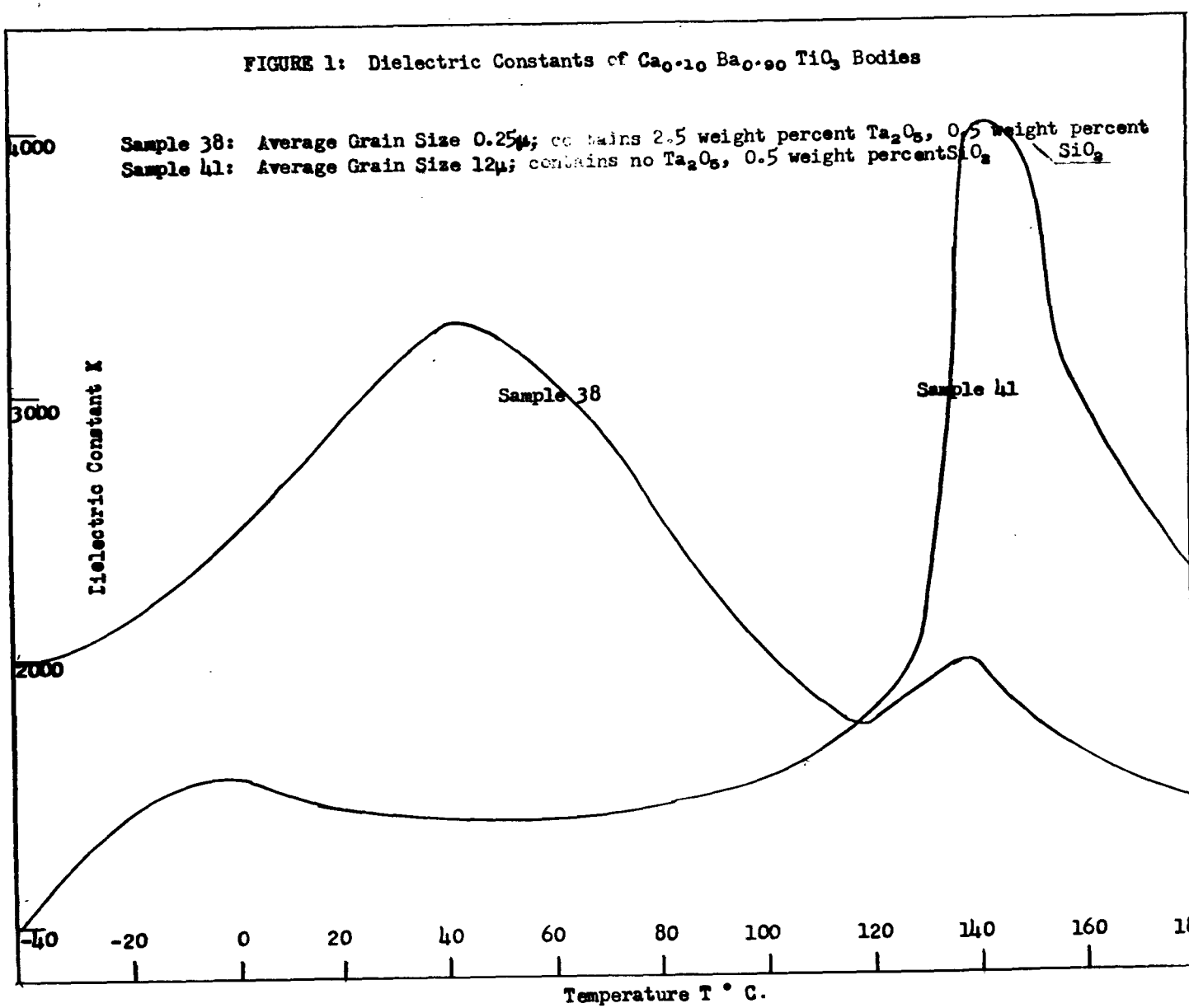
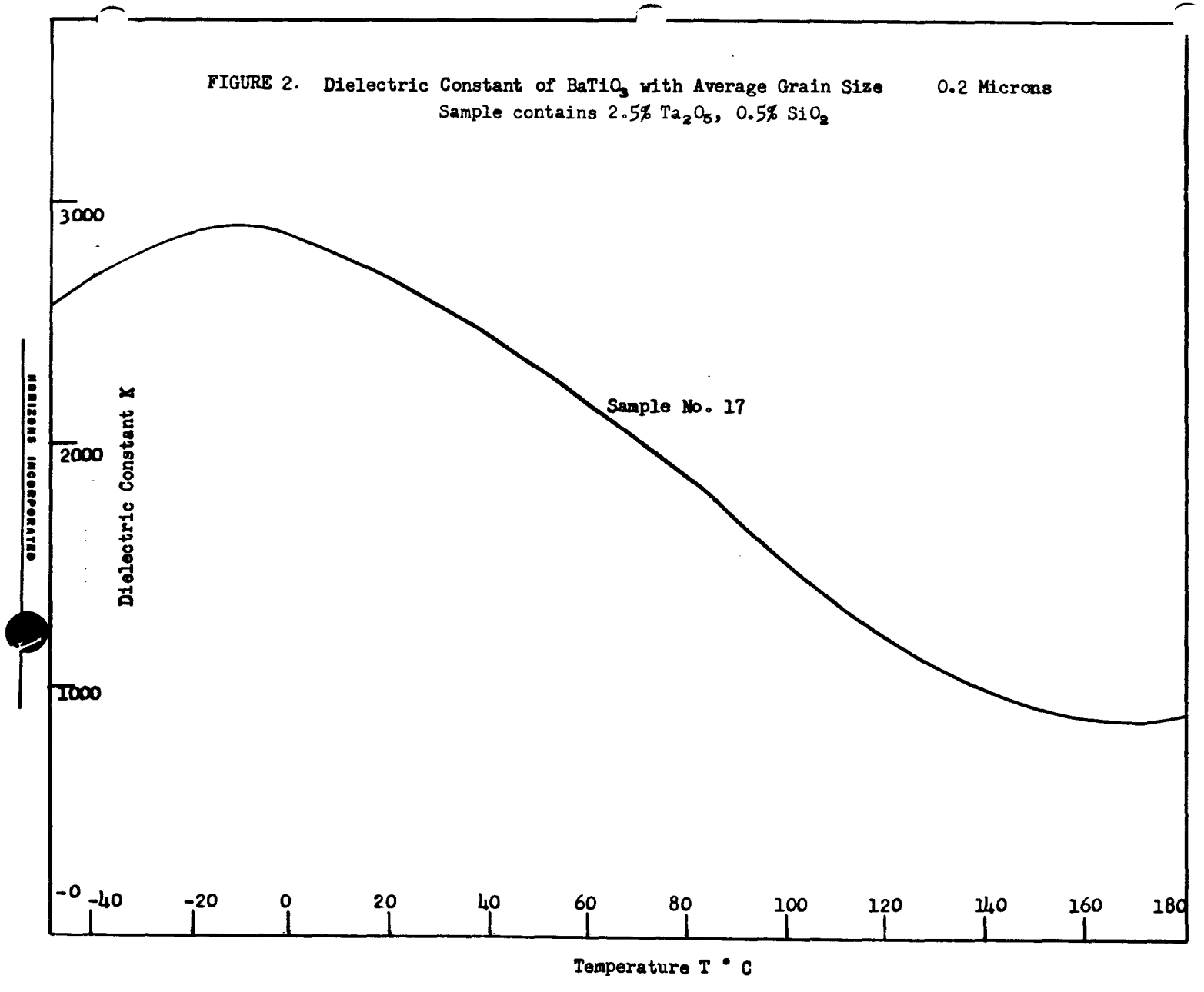


FIGURE 2. Dielectric Constant of $BaTiO_3$ with Average Grain Size 0.2 Microns
Sample contains 2.5% Ta_2O_5, 0.5% SiO_2



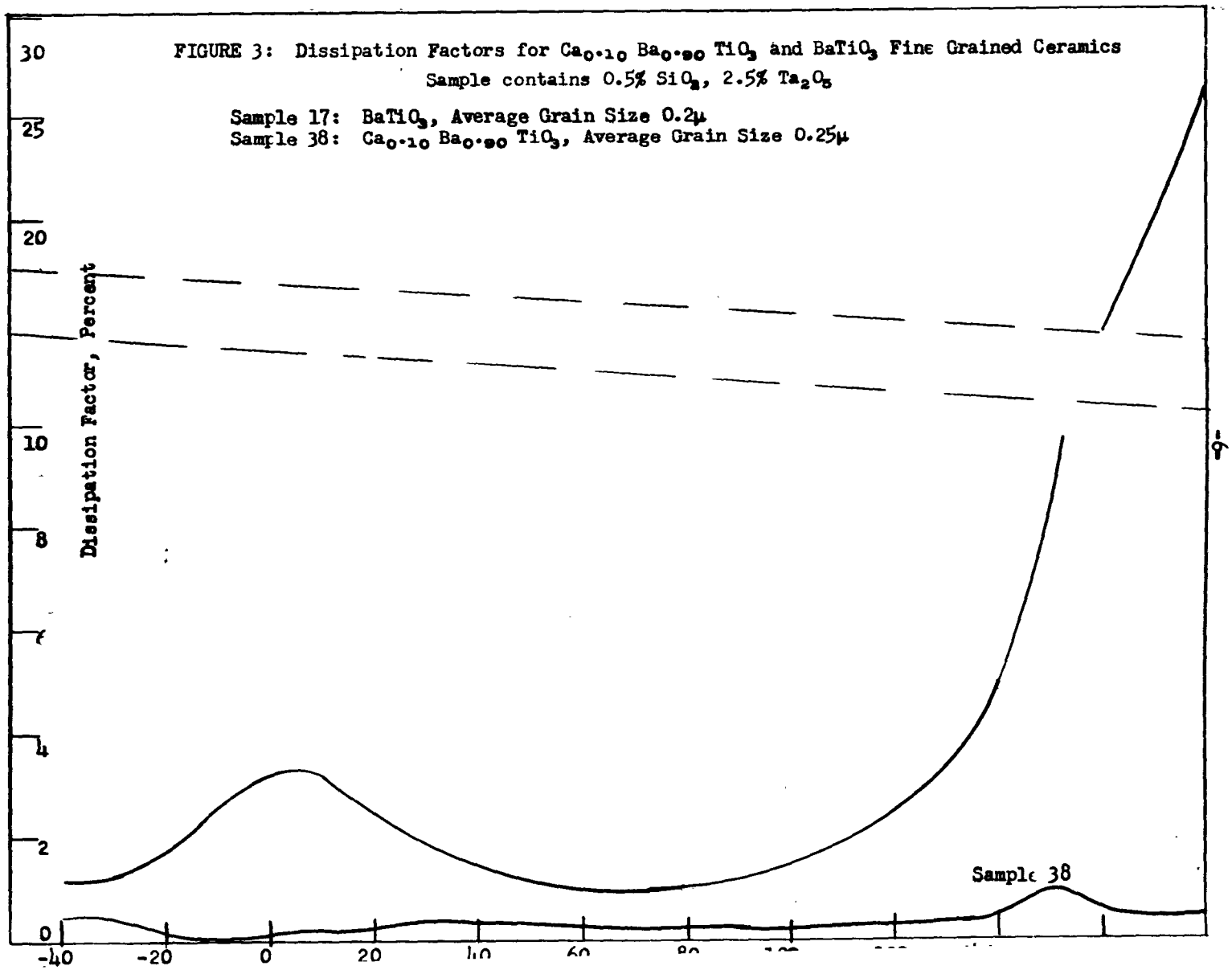


FIGURE 3: Dissipation Factors for $Ca_{0.10}$ $Ba_{0.90}$ TiO_3 and $BaTiO_3$ Fine Grained Ceramics
Sample contains 0.5% SiO_2, 2.5% Ta_2O_5

Sample 17: $BaTiO_3$, Average Grain Size 0.2μ
Sample 38: $Ca_{0.10}$ $Ba_{0.90}$ TiO_3, Average Grain Size 0.25μ

2. THIN FILM STUDIES

2.1 Objectives and General Methods of Approach

The immediate objective in this phase of the research program is to obtain thin, dense, polycrystalline films of barium titanate with a controllable grain (i.e., crystallite) size. The methods selected for consideration were more or less of general applicability, so that optimum compositions could subsequently be chosen, based on evaluations of the electrical behavior of thick ceramic bodies.

Two avenues of approach were to be followed. In the first of these, a dispersion of the particulate material of a suitable size and size distribution would be formed into a film either in or on an appropriate medium, and fired to induce sintering and densification. For example, a deflocculated slip of barium titanate may be deposited on a volatilizable film substrate, placed on a smooth refractory setter, and fired to form a dense free film.

A second approach would be to deposite a film from a solution containing suitable compounds of, for example, barium and titanium; the system is subsequently induced to react, for example, by heating, so as to produce a dense film of the dielectric material. This method (the "chemical" method) has the advantage, in principle, of permitting the achievement of much smaller crystallite sizes than the first ("ceramic" method).

2.2 Deposition of Films from Dispersions

In preliminary studies, capacitor grade barium titanate was deflocculated by stirring the material in a solution of a plastic binder in an organic solvent, in a procedure typically as follows. 10 g. of capacitor grade

$BaTiO_3$, with an average particle size about 3 microns, was stirred into a solution containing 1.5 g. of nitrocellulose (Hercules type RS 5-6, viscosity 5.4) in 10 g. of amyl acetate. This corresponds to a volume ratio of about 50 $BaTiO_3$: 50 nitrocellulose. The mixture was ground for several minutes with a glass mortar and pestle, diluted with additional amyl acetate to about 50 ml, and stirred for about 24 hours. When a drop of the dispersion was spread thinly on a glass slide and allowed to dry, microscopic examination indicated that the particles had been almost totally deflocculated. Finally, 0.15 g. of dibutyl phthalate was added as a plasticizer for the plastic binder.

To form a film, the dispersion was sprayed on to a glass slide by means of an airbrush, and allowed to dry in air at room temperature. The spraying patterns and times were adjusted so that the thickness of the dried film was 20 - 40 microns, and the variation in thickness did not exceed about 10% for a given film. To measure thickness, random sections were examined microscopically.

The film was then peeled from the glass slide, pressed between two glass plates, heated for two hours at 130°C, and then cooled. It was found that this procedure gave a strong flexible film which was flat and free from wrinkles.

To fire, a piece of the film approximately 2 cm. square was laid flat on a fired barium zirconate setter about 0.5 cm. thick by 4 cm. in diameter. The latter was inserted into a furnace preheated to 1200°F, to ignite the binder and cause a partial presintering of the barium titanate. It was found that this procedure gave a flat film of the ferroelectric material with sufficient green strength to facilitate handling without breakage.

Shrinkage during this presintering operation was about 20 - 25% along each edge.

To densify the presintered material, the film on the setter was transferred to a furnace preheated to 2500°F, and fired for 20 minutes. It was then rapidly cooled about 400F deg., and allowed to cool slowly to room temperature over a period of several hours. This annealing procedure was followed in order to relieve strains possibly caused by the volume change associated with the hexagonal-tetragonal phase inversion.

It should be noted that, starting with an unmodified capacitor grade of barium titanate, the procedure, above yields grains sizes of 3 to 10 microns, as expected from the behavior of thicker sections of barium titanate under similar firing conditions. Also, the thickness of fired film resulting from a starting thickness of 20 microns, is about 10 microns; so that in many areas the densified film is only a single grain thick.

Further work along this approach will be directed to controlling grain sizes to below 1 micron, and incorporating colloidal starting materials to duplicate the effects of close packing in order to obtain tightness. When films free from holes are obtained, complete electrical evaluations will be performed.

2.3 Chemical Methods of Film Formation

Some preliminary experiments were performed in order to determine the physical and chemical characteristics of films deposited from mixed alcoholate solutions (2), (3), as developed by U. S. Army Electronics Research and Development Laboratories. In these preparations, films were deposited on glass surfaces; their thickness and porosity were estimated, they were checked for electrical conductivity, and analysed by x-ray diffraction.

A conductive glass surface was first prepared as follows. A glass slide was heated to 1000°F, and while still in the furnace, sprayed with a solution containing equal amounts of stannic chloride, water, and ethanol. When the glass surface had just begun to aquire a whitish coating, spraying was discontinued and the glass allowed to cool slowly. The conductivity obtained was about 500 ohms per square.

In order to deposit a continuous, adherent coating of barium titanate, a solution was prepared containing the equivalent of 1% $BaTiO_3$, by mixing the appropriate amounts of tetraisopropyltitanate, an alcoholic solution of barium methoxide, and anhydrous methanol. To form the coating, the conductive glass slide was dipped into the solution, withdrawn slowly, and allowed to dry at 110°C for 1/2 hour.

In order to view the film in section, the glass slide was cut by scratching the back and breaking. By visual examination of a section with a reflecting microscope, and by studying the behavior of the interference colors of reflected light, the thickness of the film was estimated at 2000 to 5000A°.

To deposit coatings a few hundred times this thickness, a solution was prepared similar to that described above, except containing the equivalent of 5% $BaTiO_3$. This was sprayed on to the surface of a conductive glass slide, previously heated to 100°C, followed by a bakeout at 100°C for 15 minutes. In three or four spray passes, it was found possible to build up a coating thickness of 10 to 20 microns.

Glass slides thus coated were heated in order to induce conversion to polycrystalline barium titanate. It was found, however, that the coatings

charred on heating to 800°F, and could not be identified by x-ray diffraction as crystalline barium titanate until they had been fired several minutes at 1100°F. In Figure 4 are plotted x-ray diffraction intensities obtained directly from coated glass plates. Curve A represents C.P. barium titanate, as a standard deposited on a glass slide, in an ethylcellulose binder. Curves B and C represent intensities for coatings deposited from the mixed alcoholate solution, and fired 20 minutes at 1100°F and 700°F , respectively. It is evident that little or no barium titanate is detectable by this method when the firing temperature is not above 700°F.

Photographs of fractured sections of $BaTiO_3$ films fired at 1100°F, are given in Figure 5. The coatings were white, very adherent, but possessed sufficient porosity to make measurements of dielectric constant not significant. Grain sizes were estimated optically to be well below 0.5 microns. By impregnating the film and refiring it a few times, it should be possible to obtain films 10 - 20 microns thick, with the porosities reduced to a negligible level, and having densities approaching theoretical.

FIGURE 4: X-ray Diffraction Curves of Deposited Films

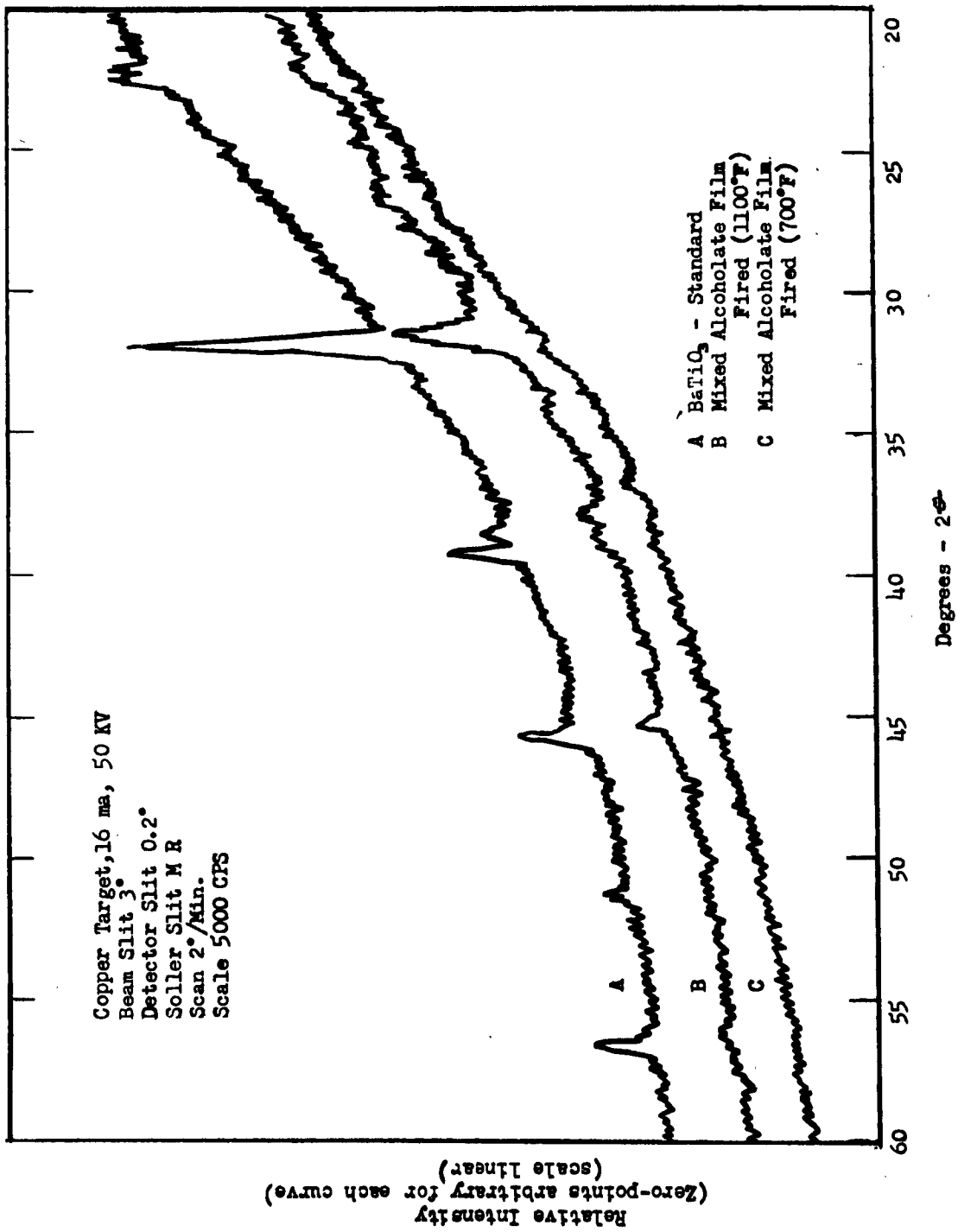


FIGURE 5: $BaTiO_3$ Films on Conductive Glass
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Fired 8 Hours at 1100°F

Fired 20 Minutes at 1100°F

3. MEASUREMENT OF PYROELECTRIC EFFECTS

Equipment has been assembled for obtaining the polarization versus temperature curves of ferroelectric materials using the pyroelectric technique (4). The sample is mounted in a brass chamber which is immersed in a silicone fluid bath whose temperature may be varied with an electric immersion heater. This chamber is equipped with a port so that radiant flux from a microscope illuminator may be utilized as a chopped thermal input source to the sample. The face of the sample facing the port is coated with Oildag in order to increase its thermal emissivity. The light source is chopped using a sector wheel driven by a General Radio d.c. variable speed motor. An adjustable regulated power supply is in series with the sample and a Tektronix Type 122 low level preamplifier. This power supply allows the polarization measurements to be carried out at different bias levels. The output signal from the preamplifier is recorded on a Brush Mark II strip chart recorder.

In order to establish that the equipment was functioning properly, a conventionally fired and polarized barium titanate specimen was mounted in the sample holder and the polarization current determined as a function of temperature. The curve obtained was essentially identical with that reported in the literature for barium titanate. A series of barium titanate densified bodies having varying amounts of tantalum additions in order to stabilize the grain size at different values are now being examined using this equipment. The initial results indicate that the polarization curve is, as expected, quite different from that of conventional barium titanate. These experiments involving the pyroelectric effect as well as other similar

experiments involving photoeffects will be completed during the next reporting period and reported in the next quarterly report.

PROGRAM FOR THE NEXT CONTRACT PERIOD

During the next period of this project, both the "ceramic" and "chemical" methods will be used, as described in this report, to produce both unsupported ferroelectric films, and films bonded to an electroded substrate. These films will be analysed directly by x-ray diffraction for phase constitution, and by x-ray fluorescence for stoichiometry. Porosity will be estimated using electrical methods (dielectric constant, dielectric strength) and physical methods (direct microscopy, chromatographic permeability).

The electrical characteristics of suitably dense films will be compared with the behavior expected from that of comparable thick densified bodies. Sub-optical grain sizes of the films will be measured by electron microscopy of direct replications of the film surfaces.

Pyroelectric measurements will be used to study polarizations in $BaTiO_3$ bodies having average grain sizes down to 2000Å, and photoeffects in these materials will also be studied.

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3) Janina J. Mitchell, "Process of Making Mixed Oxide Films" U. S. Patent 3,066,048 (U. S.-Secretary of the Army).

4) A. G. Chynoweth, J. Appl. Phys. 27, 78 (1956).

"Your comments, suggestions, and criticisms on the work reported would be appreciated. Longhand notations on this sheet will suffice. Please detach and send to the attention of Dr. E. Wainer, Horizons Incorporated, 2905 East 79th Street, Cleveland 4, Ohio."

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Eugene Wainer Director of Research	22
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3. Fine-Particled Ferroelectric Materials

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II. Fotland, R. A.
III. U. S. Army Electronics Contract DA 36-039 sc-89203

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